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VIBRATIONAL SPECTROSCOPY OF THE ELECTRODE-SOLUTION INTERPHASE. --ETC(11)
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Vibrational Spectroscopy of the Electrode-Solution Interphase.IV. FTIR: Experimental Considerations.

Stanley Pons, Timothy Davidson, and Alan Bewicks

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University of Alberta Department of Chemistry Edmonton, Alberta, Canada 76G 2G2

Department of Chemistry The University - ' Southampton, U.K.

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SUPPLEMENTARY MOTES

Electrochemistry, Spectroelectrochemistry, Reflection Spectroscopy, FTTR

ABLTBACT (Come -----

The experimental details for the gathering of high quality infrared a electrode-solution interphase are given.

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AUSTRACT

The experimental considerations accessary to effect successful recording of in situ vibrational spectroscopy at the electrode-solution interface are described. In this work, the Fourier transform infrared spectrometer is used.

VIBRATIONAL SPECTROSCOPY OF THE ELECTRODE-RECTROLYTE INTERPACE.

IV. POURIER TRANSFORM IMPRANKO SPECTROSCOPY;

EXPERIMENTAL CONSIDERATIONS

Stanley Pons and T. Davidson

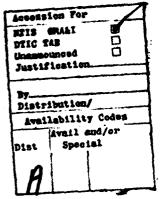
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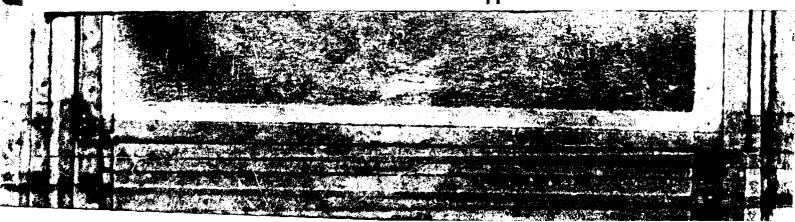
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Although the use of infrared spectroscopy as an in situ probe for observing interfacial phenomena during electrochemical experiments has been attempted many times in the last 20 years, it has only been in the last three years that high quality reproducible spectra have been routinely Obtained. This has been because of (a) the successful coupling of electrode modulation techniques to the optical signal (1), (b) the exploitation of the multiplex advantage and high throughput of Fourier transform spectrometers (2), and (c) the use of more sochisticated techniques such as polarisation modulation (3). As a result, investigations such as the study of adsorbed hydrogen on platinum and chodium (4,5), the structure of intermediates in electrocatalytic oxidations (6), the study of organic ion radical intermediates (7), the adsorption of acrylomitrile (8), the platinum interface in organic electrolytes (2,9), and the structure of water at various electrode surfaces (1,18) have been

It is the purpose of this paper to define the experimental techniques and appearatus required to obtain such results by using Pourier transform infrared methods. Although time resolved spectroscopy is possible using the techniques described herein, we will limit our discussion to mear steady-state measurements. Thus the basic approach is to apply a potential stap to an electrode, unit for steady state conditions to develop, and take one or more rapid infrared spectra. The potential is then returned to a base potential, and the procedure repeated. The entire cycle is repeated and the spectra are signal averaged to the desired signal-to-moise level. Appropriate ratioing of the 2

sets of spectra with subsequent subtraction gives the desired result.

EXPERIMENTAL.

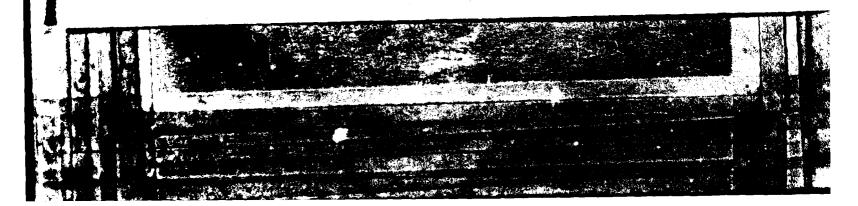
Spectrometer

The instrument used in our recent work has been the Nicolet 7199 Pourier transform infrared spectrometer. To perform these experiments, the machine should have the following features:

- (1) The ability to store, and signal average subsequent spectra, in at least three separate data files. The software should be flexible enough to specify which file is to be used for a given spectrum.
- (2) An output trigger pulse (preferably TTL or CNOW compatible) designating the beginning of data collection by the A/D Converter of the instrument On each scap.
- (3) Spectral ratioing and subtraction capability.
- (4) Capability to handle mid-IR fast detectors with minimum D⁰ values of 1 \times 10¹⁰, such as Mercury-cadmium-talluride 77 K solid state units.
- (5) Sample compartment sizes large enough to hold comparatively large cells mounted on adjustable reflection attachment optical tables.
- (6) Reasonably fast data collection times.
- (7) Resolution of 8 cm⁻¹ or better.

Data Presentation

The most convenient form in which to present meetra is as a difference in infrared transmission of the cell at each of the



two electrode polarization states. This difference will then be due to changes in the interphase brought about by the differences in potential, the optical properties of the rest of the cell remaining constant. In terms of the measured reflectivities at the two potentials, R_1 and R_2 , this difference in transmission can be written as R_2/R_1-1 , a quantity which is readily obtained using software normally provided with connercial FTIR spectrometers. In practice, it is most convenient to calculate R_2/R_1-1 as $R_2/R_1-R_3/R_1$. This latter quantity is simply the difference between two single beam spectra, each of which has been ratioed to the chosen reference state R_1 . Presentation of the spectra in this form is thus equivalent to the $\Delta k/R$ presentation used in EMIRS ($\Delta R/R$ is equivalent to $(R_2-R_1)/R_1$).

Electrochemical Instrumentation

The potential was controlled at the working mirror electrode by a Hi-Tek Instruments DT2101 potentioetat driven by a PPR1 waveform generator. The Nicolet computer supplied a TTL pulse to a SMC connector on the rear punel as each new scen was initiated. This pulse was used to trigger the waveform generator via a 74190 TTL up-down counter. (See discussion.)

Electrodes and Cell

Electrodes (Chemical Electronics Associates, 333 slouth County Road, P.O. Box 3126, Pain Beach, Fig.) were constructed from a 2 mm thick = 7 mm diameter disk of the desired material effixed to the end of a 7 mm \times 135 mm brans rod which had been heat/press fitted into a Kel-F sleeve (o.d. = 9 mm). A loose fitting Kel-F sleeve at the rear of the electrode assembly was

made to butt a rubber gasket against the fixed sleeve by a mut at the end of the assembly to seal it into the cell barrel. The brass rod was covered with a thin layer of epoxy before sealing it into the Kel-F sleeve to prevent any chance of leakage of solution between the electrode and the sleeve. Electrodes prepared in this manner are very stable mechanically and chemically in most solvents and electrolytes for several months. For instance, we have used 1 M sulfuric acid, 1 M perchloric acid, acetonitrile, methylene chloride, dimethylfornamide, dimethylecetamide, and dichloroethame for extended periods without degradation of electrode performance.

Electrodes are typically polished by mounting in a sturdy aluminum jig to maintain perpendicular contact with the polishing pad, or on a commercial polishing machine. With reasonable care, electrodes flatter than 1/2 at 5 microns were obtainable using these rather simple techniques.

Even though the electrodes are reasonably flat, it is necessary to take additional care to assure that the face is parallel to the optical window, so that a uniform solution layer thickness will exist there. This is accomplished by allowing the optical window to be bonded to its ground glass sleeve with both sleeve and electrode in place in the cell and with the window resting on the face of the mounted electrode, pressure being maintained against the window simultaneously to keep it absolutely flat against the electrode face (Figure (1)). The electrode, window cap, and cell body were acribed to maintain reproducible realignment in subsequent experiments. When the window was prepared in this manner, typical fluctuations in gap



size of 20.05 microns were obtained in measurements from side to side across the electrode face using the integrated extinction coefficient of the acetonitrile CIM stretch fundamental shaperstion band as a measure of the cap thickness.

The cell (Chemical Electronics Associates, Inc.) was similar to that described by Bewick et al for use in electrochemically modulated infrared reflectance spectroscopy (ENINS) (11). It was 125 mm in overall length without the electrode mounted, and held 50 cm³ of solution. The optical window was mounted on a T 24 female ground glass joint. When in place, the working electrode disk may be pushed up flush to the window. A Luggin reference capillary was mounted with its tip close to the window where the electrode face rests. The Luggin is connected through a ground glass stopcock to the reference electrode compartment. A platinum wire loop was mounted such that the loop was consist with the mounted working electrode to serve as the secondary electrode. Purge gas inlet and outlet connections were made at the bottom and top of the main cell body, and the working electrode mounting tube was located at the rest.

A small pistinum wire to be used as a subsidiary reference electrode may be mounted through the Luggin tube just below the stopcock to provide a low impedence path and reduce high frequency interference. When used, it was connected through a 8.1 pF capacitor to the normal reference electrode,

Reflectance Attachment

The converging beam of radiation through the sample compartment of the spectrometer must be interrupted and made to

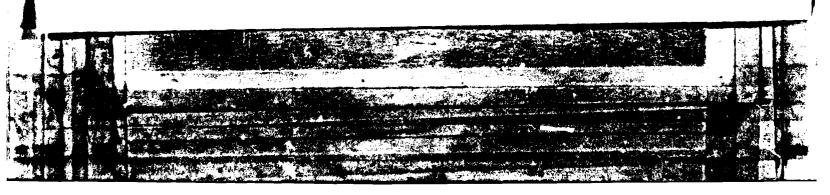
reflect from the electrode surface at a dic red angle. Subsequently, the beam must be returned to its original path as closely as possible so as not to affect the purpose of the focussing optics of the output detector section of the spectrometer. Several designs for an apparatus to perform this function are possible. We have used a modified version of an apparatus described by Marrick (12).

Polarization of the radiation was effected by mounting a KBr substrate transmission polarizer (Cambridge Physical Sciences, Cambridge, England, Type IGP228) in the beam either in front of or behind the cell. The polarizer was mounted in a calibrated housing that could be rotated so that any polarization angle could be conveniently chosen.

Solvents and Chemicals

Due to the extremely high sensitivity of the method, it is necessary to use very pure solvents and electrolytes for routine investigations. We have found that the use of MPLC grade commercial solvents are suitable for most organic intermediate studies (7), while further purification of these solvents in usually required for sensitive adsorption or double layer studies (9).

The drying of supporting electrolytes, such as lithium perchlorate or tetraelkylammonium selts should be effected with a vacuum oven for at least 24 hours at the maximum possible temperatures. Transfers of electrolytes and solutions to the cell should be made in a dry atmosphere.



MESULTS AND DISCUSSION

As an example of a typical experiment, it was desired to record the spectrum of the benzophenome ketyl species formed by the one electron reduction of benzophenome at a platinum electrode. A 10 mM solution of benzophenome in degassed anhydrous 0.10 <u>M</u> tetrabutylammonium tetrafluoroborate was used. The electrode was placed flush against the cell window, and allowed to relax against the rubber gasket on the electrode assembly. Using the 2220 cm⁻¹ acetomitrile band, the solution thickness was found to be about 5 microns, as determined by the relation

4 - 2A COSS.

d is the solution thickness, A is me "morbance, a is the extinction operation, C is the concentration of the absorbing species, and 0 is the angle of incidence at the reflecting surface. The polariser was set to pass the component of light that had its electric field vector parallel to the incidence plane, so that the field would have a non-zero magnitude at the electrods surface.

The "begin scan" TTL pulse available from the computer of the PTIR spectrometer was used as input to a 74190 binary up counter whose binary output had initially been preset to binary output 15 (iiii). The MSB output of the counter was connected to the "start" TTL input of the PTR1 waveform generator so that the latter executed one waveform sequence on every 0 + 1 transition from the counter, i.e. every 16 scans of the spectrometer. The waveform generator was set to give a rectangular pulse f 9 s duration (about 1 second longer than the spectrometer requires to scan 8 spectra) such that the electrode potential was taken to -2.50 V (Ag/Ag* reference) for 9 s then returned to the base value of -1.70 V until the spectrometer initiated the 17th scan and the complete sequence was repeted.

A software program was written to perform eight scans, of which only the last four were co-added and saved in a reference file. This or station allowed about four seconds (the first four unused scans) for the cell to come to a steady state. The program then called for eight more scans to be made, again discarding the first four, but this time storing the co-added last four in a sample file. This operation was repeated 10 times so that a total of forty co-added spectra resulted in the reference file and the same in the sample file. This program was interfaced synchronously to the waveform generator through the "begin scan" pulse and the 76190 counter circuitry. Thus the spectra were gathered at the approximate points indicated in Figure (2), An example of the spectrum computed from this data in the manner described in the experimental section is shown in Figure (3).

Basically, the peaks extending upwards correspond to the removal of bensophenone substrate, at the expense of the formation of betyl (downward extending peaks). A full description of this spectrum has been reposted (7).

The spectra in Figures (4) and (5) were obtained in a similar manner. Details are provided in the figure legends.

Higher sensitivity was needed to observe changes in the population of species as the double layer structure was changed at a platinum electrode. A thinner solution layer (0.75 microns) and 100 averaged spectra at each potential state was utilized. The electrolyte was 0.10 M tetrabutylammonium tetrafluoroborate. The resulting difference apectra are shown in Figure (6). The effect of added water for the same experiment is shown in Figure (7). These spectra have been interpreted elsewhere (6).

It is clear from the spectra shown that the technique has the sensitivity to detect changes approaching 10⁻⁶ AR/N in experimer's collecting data from 100 scans at each potential, the total experimental time being about 10 min. Thus the method seems well suited for the obtaining of vibrational spectra at the electrode-solution interface in situ, and should be suitable for the structural determination of a variety of catalytic, adsorbed, and short-lived solution-free intermediates formed at such interfaces.

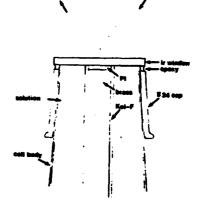
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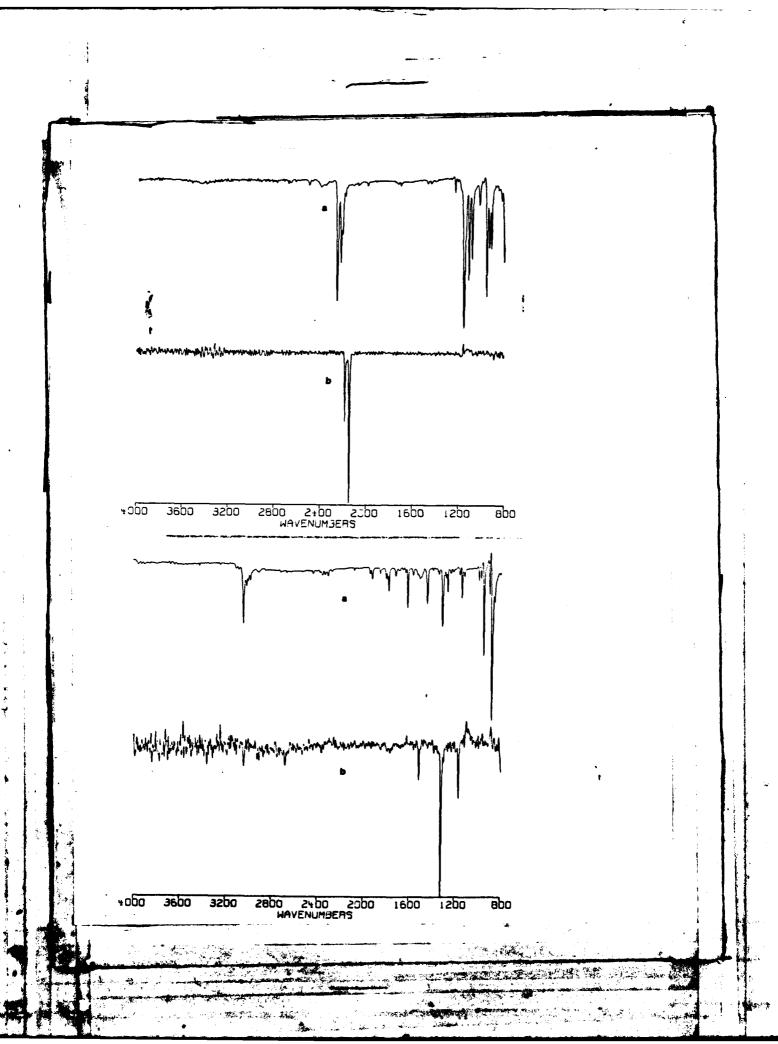
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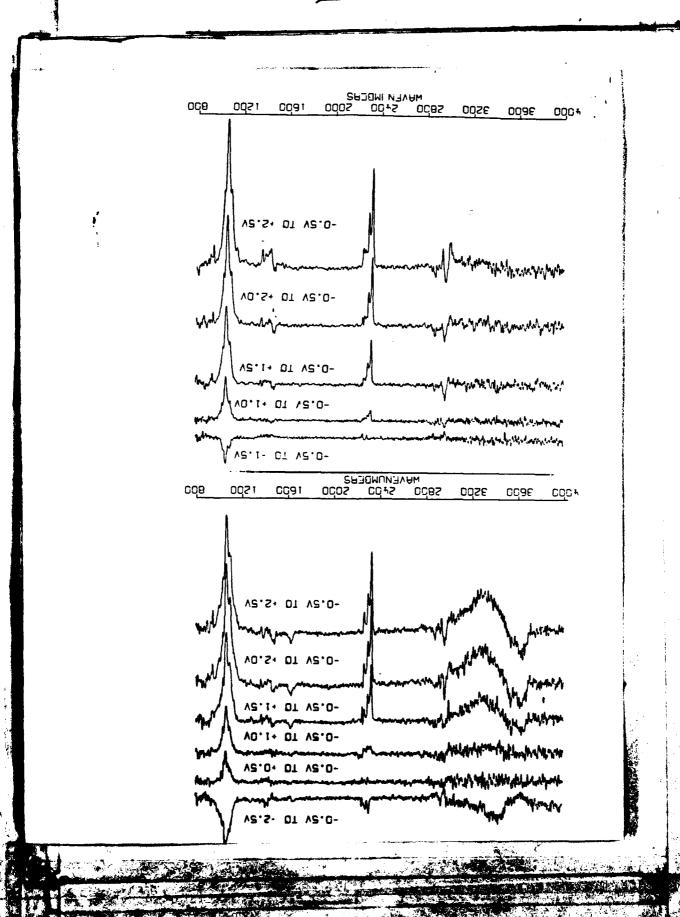
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- (1) Cell cap and electrode tip configuration.
- (2) Timing for spectra acquisition. Points (d) represent spectra that are discarded, points (s) represent spectra that are saved and co-added. Diagram is for case where more absorbing spectra are present at high sampling potential.
- (3) a) Transmission spectrum of bensophenone. (b) Difference spectrum between -0.75 V and -2.50 V (lu mM bensophenone in 0.10 M TBAF in acctonitrile). Downward extending yeaks are for the ketyl anion.
- (4) a) Transmission spectrum of anthracene. (b) Difference spectrum between -1.50 V and -2.50 V.
- (5) a) Transmission spectrum of tetracyanoethylene. (b) Difference spectrum between +0.25 and -0.25 V.
- (6) Difference spectra of ambydrous 9.10 $\underline{\text{M}}$ tetrabutylammonium tetrafluoroborate.
- (7) Same as Figure 6, except 8.10 $\underline{\mathrm{H}}$ N₂O includes.







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